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⑮ Liquid detergent compositions.

⑮ Aqueous liquid detergent compositions are required to be non-corrosive to the vessels in which they are used and have viscosities allowing convenient use. Use of amorphous silicas having defined surface area, mean pore diameter, oil absorption and weight mean particle size as components allow these desired properties to be achieved.

EP 0 353 075 A2

A

## Description

## LIQUID DETERGENT COMPOSITIONS

## FIELD OF THE INVENTION:

5 The present invention relates to physically stable liquid detergent compositions and in particular to aqueous liquid detergent compositions having non-corrosive properties and satisfactory viscosities.

## BACKGROUND TO THE INVENTION:

10 Aqueous liquid detergent compositions, particularly those containing sodium tripolyphosphate corrode the enamel and aluminium parts in washing machines, and this effect is exacerbated by the presence of perborate or percarbonate salts. It is desirable to include an anti-corrosion agent in the detergent composition to inhibit the corrosive action so as to prevent the formation of corrosion products that could discolour the fabric in the wash and to prolong the useful life of the aluminium and enamel parts in the washing machine.

15 EP 0110472 (Unilever) discloses that the corrosive action of aqueous liquid detergent compositions can be reduced to a significant extent by incorporation of silica, usually from 1% to 10% w/w, of such type and in such amount that in the wash liquor a certain threshold concentration of dissolved silica is established. It was found that in order to achieve optimal corrosion protection the silica must be present in the wash liquor in a dissolved form at a concentration of at least 120 ppm by weight and preferably at least 150 ppm by weight.

## 20 GENERAL DESCRIPTION OF THE INVENTION:

Liquid detergent compositions desirably have viscosities which are not excessive so that they may be efficiently handled and dosed into washing machines. The applicants have found the detergent compositions described in EP 0110472 using commercially available silicas with a BET surface area below  $550\text{m}^2/\text{g}^{-1}$  have relatively high viscosities and compositions having reduced viscosity, i.e. at the level accepted as commercially desirable preferably up to about 1000 mPas, are formed by using amorphous silicas having specific physical characteristics. Viscosities above the level quoted give rise to difficulties in handling and dosing.

25 The amorphous silicas used in the present invention have a BET surface area in the range from about 200 to about  $550\text{m}^2/\text{g}$ , a mean pore diameter from about 1.5, preferably 3, to about 11 nm, an oil absorption from about 40, preferably from about 70, to about 140 cc/100g and a weight mean particle size from about 1, preferably from about 5, to about 30 microns, preferably to about 20 microns. Preferably these silicas are incorporated at a level from about 1% to about 5% w/w.

30 These amorphous silicas provide the desired corrosion inhibition even at relatively low concentrations of about 2% to about 4 % w/w and at these concentrations an acceptable viscosity increase in the formulation is obtained. Preferably the silica added increases the viscosity of the base composition by not more than 50% preferably not more than 40% at a level of 5% w/w.

35 Suitable forms of amorphous silica are precipitated silicas and silica gels, such as hydrogels and xerogels. Precipitated silicas are preferred.

40 The precipitated amorphous silicas used in the present invention do not give a defined pore volume to nitrogen. As a consequence, the porosity present can only be defined by oil absorption and/or mercury intrusion volume within a stated range of pore diameters. In general mercury porosimetry is useful from about 3.0 to about 1000 nm.

45 For some silicas of the invention and comparison silicas it is necessary for the pore volume to be measured by the nitrogen B.E.T. Method described in Brunauer et al J. Am. Chem. SOC. Vol 60 p309 (1938). This method depends on the condensation of nitrogen into pores of silica and it is useful for measuring pores with diameters up to about 60 nm.

For certain silicas the only method of establishing the pore size distribution present is to use a combination of nitrogen adsorption and mercury porosimetry.

## COMPONENTS OF THE COMPOSITIONS:

50 The liquid detergent compositions of the invention further comprise as essential ingredient an active detergent material, which may be an alkali metal or alkanolamine soap of  $\text{C}_{10}\text{-C}_{24}$  fatty acid, including polymerized fatty acids, or an anionic, nonionic, cationic, zwitterionic or amphoteric synthetic detergent material, or a mixture of any of these. The anionic synthetic detergents are synthetic detergents of the sulphate- and sulphonate-types. Examples thereof are salts (including sodium, potassium, ammonium and substituted ammonium salts, such as mono-, di- and tri-ethanolamine salts) of  $\text{C}_9\text{-C}_{20}$  alkyl benzene sulphonates,  $\text{C}_8\text{-C}_{22}$  primary or secondary alkane sulphonates,  $\text{C}_8\text{-C}_{24}$  olefin sulphonates, sulphonated polycarboxylic acids, prepared by sulphonation of the pyrolyzed product of alkaline earth metal citrates, eg. as described in British Patent Specification No. 1 082 179,  $\text{C}_8\text{-C}_{22}$  alkyl sulphates  $\text{C}_8\text{-C}_{24}$  alkyl polyglycol ethersulphates, (containing up to 10 moles of ethylene oxides); further examples are described in "Surface Active Agents and Detergents" (vol. I and II) by Schwartz, Perry and Berch.

60 Examples of nonionic synthetic detergents are the condensation products of ethylene oxide, propylene oxide and/or butylene oxide with  $\text{C}_8\text{-C}_{18}$  alkylphenols,  $\text{C}_8\text{-C}_{18}$  primary or secondary aliphatic alcohols,  $\text{C}_8\text{-C}_{18}$  fatty acid amides; further examples of nonionics include tertiary amine oxides with one  $\text{C}_8\text{-C}_{18}$  alkyl chain and

two C<sub>1</sub>-C<sub>3</sub> alkyl chains. The above reference also describes further examples of nonionics.

The average number of moles of ethylene oxide and/or propylene oxide present in the above nonionics varies from 1 to 30; mixtures of various nonionics, including mixtures of nonionics with a higher degree of alkoxylation, may also be used.

Examples of cationic detergents are the quaternary ammonium compounds such as alkyl dimethyl ammonium halogenides.

Examples of amphoteric or zwitterionic detergents are N-alkylamino acids, sulphotetaines and condensation products of fatty acids with protein hydrolysates, but owing to their relatively high cost they are usually used in combination with an anionic or a nonionic detergent. Mixtures of the various types of active detergents may also be used, and preference is given to mixtures of an anionic and a nonionic detergent-active compound. Soaps (in the form of their sodium, potassium, and substituted ammonium salts) such as of polymerized fatty acids, may also be used, preferably in conjunction with an anionic and/or a nonionic synthetic detergent.

The amount of the active detergent material varies from about 1 to about 60%, preferably from 2 to 40% and particularly preferably from 5 to 30% by weight. When a soap is incorporated, the amount thereof is from 1 to 40% by weight.

The liquid compositions of the invention preferably also comprise up to 60% of suitable builder materials, such as sodium, potassium and ammonium or substituted ammonium pyro- and tri- polyphosphates, -ethylenediamine tetraacetates, -nitrilotriacetates, -ether polycarboxylates, -citrate, -carbonates, -ortho-phosphates, zeolites, carboxy-methyloxysuccinates. Particularly preferred are the polyphosphate builder salts, nitrilotriacetates, citrates, zeolites, and mixtures thereof. In general the builders are present in an amount of from about 1 to about 60%, preferably from 5 to 50% by weight of the final composition.

The amount of water present in the detergent compositions of the invention varies from 10 to 70% by weight.

Other conventional materials may be present in the liquid detergent compositions of the invention, for example sequestering agents, such as ethylenediamine-tetraphosphonic acid and phosphonates such as those sold under the Dequest Trade Name of Monsanto; non-builder electrolytes, such as alkalimetal-chlorides, -bromides, -nitrates and -sulphates; soil-suspending agents, such as sodium carboxymethylcellulose, polyvinylpyrrolidone or the maleic anhydride/vinylmethylether copolymer; hydrotropes; dyes; perfumes; alkaline materials, such as silicates; optical brighteners; germicides; anti-tarnishing agents; suds boosters, suds depressants, such as liquid polysiloxane anti-foam compounds; enzymes, particularly proteolytic enzymes, such as the commercially available subtilisins Maxatase (ex Gist-Brocades N.V., Delft, The Netherlands), Alcalase, Esperase and Savinase (both ex Novo Industri A/S, Copenhagen, Denmark), amylolytic, cellulolytic and lipolytic enzymes such as Lipolase (Trade Mark ex Novo) enzymes; enzyme stabilising systems, such as mixture of a polyol with boric acid or an alkalimetal borate; oxygen liberating bleaches, such as sodium perborate or percarbonate, diperisophthalic anhydride with or without bleach precursors, such as tetraacetyl ethylene diamine; or chlorine liberating bleaches, such as dichlorocyanurate; anti-oxidants, such as sodium sulphites; opacifiers; fabric softening agents; stabilisers, such as polysaccharide hydrocolloids, eg partially acetylated xanthan gum, commercially available as "Kelzan" (ex Kelco Comp., New Jersey, USA); buffers and the like.

#### TEST METHODS:

##### i) Aluminium corrosion test

In order to assess the corrosive action of a test wash liquor, circular plates of pure aluminium (99.5% Al) having a surface area of 40 cm<sup>2</sup> were cleaned to remove surface grime and contaminants by immersing the disc in nitric acid, rinsing with distilled water and air-drying. After being weighed the plates were subjected to the test liquor for a period of 18 hours at 90°C, during which time the solution was stirred continuously. Subsequently the plates were cleaned and dried using the above procedure and reweighed. The corrosive action of the test liquor was expressed in weight loss of aluminium per m<sup>2</sup> and per hour.

In all tests the liquid detergent composition was added to distilled water at 90°C at a concentration of 10 grams/litre, unless stated otherwise, and mixed using a 2.54 cm castellated stirrer for 5 minutes at 3000 rpm.

##### ii) Enamel Corrosion Test:

The test method employed is described in ISO 4533 in which the standard test solution is replaced with the aqueous liquid detergent composition at a concentration of 10g/l, and the apparatus utilised in ISO 4535.

##### iii) Surface Area:

Surface area is determined by standard nitrogen adsorption methods of Brunauer, Emmett and Teller (BET) using a single point method with a Sorptly 1750 apparatus supplied by Carlo Erba company of Italy. The sample was outgassed under vacuum at 270°C for 1 hour before measurement. The pore volume for silicas having pore diameters below about 3 nm was determined using the adsorption isotherm obtained.

## iv) Oil Absorption:

The oil absorption is determined by the ASTM spatula rub-out method (American Society of Test Material Standards D, 281).

The test is based upon the principle of mixing linseed oil with a silica by rubbing with a spatula on a smooth surface until a stiff putty-like paste is formed which will not break or separate when it is cut with the spatula. The volume of oil used is then put into the following equation:-

$$\begin{aligned} \text{Oil absorption} &= \frac{\text{cm}^3 \text{ oil absorption} \times 100}{\text{wt. of silica sample in gms}} \\ &= \text{cm}^3 \text{ oil/100g silica} \end{aligned}$$

## v) Weight Mean Particle Size:

The weight mean particle size of the silicas was determined with the aid of a Malvern Particlesizer, Model 3600 E. This instrument, made by Malvern Instruments, Malvern, Worcestershire uses the principle of Fraunhöffer diffraction utilising a low power He/Ne laser. Before measurement the sample was dispersed ultrasonically in water for a period of 7 minutes to form an aqueous suspension.

## vi) Mean pore diameter (MPD):

This parameter is related to the surface area and pore volume and, using a cylindrical pore model, is calculated for a silica product with the formula

$$\text{MPD (nm)} = \frac{\text{pore volume (cc g}^{-1}\text{)} \times 4,000}{\text{surface area (m}^2\text{g}^{-1}\text{)}}$$

Pore volume is measured using mercury intrusion method (viii) or nitrogen adsorption (iii).

## vii) Viscosity:

This was determined on a liquid detergent composition which has been allowed to stand for at least 48 hours after preparation. A Haake Rotovisco RV12 was used under the conditions:

measuring head	M500
sensor system	MVI
speed 8 rpm	shear rate 19 s <sup>-1</sup>
temperature	25°C

Viscosity measurement was taken after 2 minutes rotation of the bob.

## viii) Mercury intrusion volume:

Mercury intrusion volumes are determined (in cc/g) by standard mercury intrusion procedures using a Micromeritics Autopore 9220 mercury porosimeter. The pore radius is calculated from the Washburn equation using values of surface tension for mercury of 485 dynes/cm and contact angle of 140°.

Prior to measurement the sample was outgassed at room temperature to a pressure of 50 microns of mercury. The mercury intrusion volume recorded is that occurring over the range of calculated pore diameters of 0.05 to 1.0 micron.

SPECIFIC DESCRIPTION OF THE INVENTION:

The invention is further illustrated but not limited by the following Examples, in which parts and percentages are by weight, unless indicated otherwise.

Tables I and II quote silica product and test characteristics. The silicas are indicated by letters which correspond to the following products.

A.)		
B.)	Precipitated silicas available from Crosfield Chemicals of Warrington England and within EPA 0308165	5
C.)		
D.)		
E.)		10
F.)		
G.*	Gasil 200 TP within EP 0110472.	
H.	Silica within EPA 0227334 obtainable from Crosfield Chemicals.	15
I.*	Microsil GP.	
J.*	Sorbsil C60-10	
K.*	HP25	20
L.**	Zeo 49B	
M.	Silica gel prepared according to GB 2038303 (Grace)	25
* Comparison silicas obtainable from Crosfield Chemicals, Warrington, England.		
** Comparison silica obtainable from J M Huber Corporation, Havre de Grace, Maryland, USA.		

The silicas labelled A, B, C, D, E, F, H & M are silicas used in the invention, Silica M was prepared using the Example 2 test 10 conditions of GB 2038303 followed by fast drying and air micronisation.

Gasil, Microsil, Crosfield HP and Sorbsil are registered trademarks of Crosfield Chemicals, and Zeo is a registered trademark of J M Huber Corporation.

Two base detergent compositions similar to the formulations disclosed in EP 0110472 have been used to illustrate the invention. Formulation A contains all the ingredients listed except for the silica, and the water balance is adjusted to allow the silicas to be added as a 20% w/w aqueous slurry in the final act of preparing the detergent composition for test.

In formulation B, the silica is added as an ingredient at the preferred place in the formulation usually before the addition of the non-ionic surfactant and the water balance adjusted accordingly.

It must be emphasised that the viscosity of the detergent composition varies with each preparation, and so the percentage increase or decrease compared to the control is the only method which can be used to monitor change.

The preferred use of detergent composition A is to screen silicas and absolute determination of product characteristics are usually only performed when the silica is incorporated into the detergent composition at the preferred place of addition, i.e. using composition B.

#### EXAMPLE I

The silicas as listed in Table I were dispersed at 20% w/w loading into detergent composition A as aqueous slurries at ambient temperature using a castellated stirrer (diameter 2.54 cm) at approximately 1000 r.p.m. for 2 minutes. The viscosity and corrosion inhibition of each test formulation was determined using the test methods described above.

The results shown in Table II demonstrate that only silicas A, B, C, D, E, F, H, M and EP 0110472 silica G (surface area 750 m<sup>2</sup>/g) give the desired small increase in viscosity coupled with corrosion inhibition. The latter is a silica with a surface area above the level at which the viscosity problem identified by the applicants occurs.

TABLE I

EXAMPLE/ PROPERTY	SURFACE AREA (m <sup>2</sup> /g)	PORE VOLUME (cc/g)	MEAN PORE DIAM (nm)	OIL ABSORP. (cc/100g)	WEIGHT MEAN PARTICLE SIZE (microns)	PH	SO <sub>4</sub> <sup>=</sup> % w/w	Cl <sup>-</sup> % w/w
SILICA A	461	0.46*	4.0	115	10.2	5.6	0.73	0.45
SILICA B	450	0.30*	2.7	94	11.5	4.2	2.76	1.78
SILICA C	522	0.42*	3.2	110	14.4	6.0	0.13	0.20
SILICA D	522	0.42*	3.2	110	9.7	6.0	0.13	0.20
SILICA E	522	0.46*	3.5	115	3.5	6.0	0.13	0.20
SILICA F	507	0.18*	1.4	75	8.8	6.7	0.09	0.10
SILICA G	750	0.38+	0.2	70	8.9	3.8	0.61	0.08
SILICA H	250	0.64*	10.2	130	8.0	6.9	0.12	0.10

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TABLE I (Continued)

SILICA I	266	1.70*	25.6	290	18.3	6.3	0.98	0.08
SILICA J	500	0.75+	6.0	200	4.7	6.5	0.05	0.08
SILICA K	400	1.0+	10.0	172	5.3	7.0	0.50	0.10
SILICA L	241	1.07*	17.8	94	8.9	7.5	0.35	0.12
SILICA M	414	0.17+	1.6	40	16.0	2.5	2.15	0.04

\* pore volume determined by mercury porosimetry

+ pore volume determined by nitrogen adsorption



TABLE II

EXAMPLE/TEST	VISCOSITY (mPas)	% VISCOSITY INCREASE VS CONTROL	CORROSION WT. LOSS OF ALUMINIUM (g/m <sup>2</sup> /h)
SILICA A	644	19.3	0.005
SILICA B	652	20.9	0.002
SILICA C	623	15.4	0.009
SILICA D	644	19.3	0.009
SILICA E	558	3.3	0.009
SILICA F	661	22.4	0.007
SILICA G	653	20.9	0.002
SILICA H	636	17.8	0.009
SILICA I	876	62.2	0.003
SILICA J	716	32.6	0.070
SILICA K	699	29.4	0.006
SILICA L	725	34.3	1.49
NO SILICA	540	CONTROL	1.79
SILICAM	739	5.0	0.003
NO SILICA M	704	CONTROL	1.79

TABLE III

SILICA	DET. COMPOSITION 2% w/w SILICA		DET. COMPOSITION 3% w/w SILICA		DET. COMPOSITION 4% w/w SILICA		DET. COMPOSITION 5% w/w SILICA	
	VISC. mPas	VISC. INCREASE %	VISC. mPas	VISC. INCREASE %	VISC. mPas	VISC. INCREASE %	VISC. mPas	VISC. INCREASE %
G	713	2	807	16	910	31	1013	46
A	730	5	842	21	970	40	1039	49
H	721	4	833	20	945	36	N-M	N-M
I	927	33	1245	79	N-M	N-M	N-M	N-M

DETERGENT COMPOSITION CONTAINING NO SILICA - VISCOSITY 696 mPas.

N-M - not measured because 20% silica slurry has excessive viscosity.

EXAMPLE II

Silicas A and H, together with two comparison silicas G and I, were dispersed at loadings of 2% to 5% w/w into detergent composition A as 20% w/w aqueous slurries at ambient temperature using the same dispersing conditions as in Example I. The viscosity was determined on each test/formulation according to the method described above. Silicas H and I could not be loaded to a concentration of 5% w/w and 4% w/w respectively, owing to excessive viscosities in the aqueous slurry. The results are summarised in Table III and they clearly show the silicas A and H used in the invention give a similar performance for viscosity increase against loading up to 4% w/w, as comparison silica G which has a surface area in the preferred range as disclosed in EP 0110492. It can also be seen that the commercially available low surface area silica I thickens the detergent composition excessively.

EXAMPLE III

Silica A was dispersed into detergent composition B, prior to adding the non-ionic surfactant, at a loading of 2% w/w. Before addition, the silica was dispersed in water at a concentration of 20% w/w. Three preparations of the detergent composition containing the silica used in the invention were prepared, together with three compositions where the silica was omitted and the water balance adjusted accordingly. The viscosity of each preparation was determined according to the method described above. The results are summarised in Table IV. The data shows that the viscosity of the detergent composition does vary with each preparation, but for the formulations containing the silica used in the invention the viscosity increase is not greater than 25% in each case.

TABLE IV

PREPARATION	VISCOSITY (mPas) DETERGENT COMPOSITION WITHOUT SILICA	VISCOSITY (mPas) DETERGENT COMPOSITION WITH SILICA A	% VISCOSITY INCREASE
I	618	723	17
II	642	792	23
III	710	842	19

EXAMPLE IV

In the examples given above it has been demonstrated that silicas used in the invention are capable of inhibiting the corrosive action of the detergent composition without causing undesirable increases in viscosity of the formulation. For commercial benefit, these desirable features need to remain in the detergent composition for a period of at least three months.

Silicas A and F together with comparison silica G were dispersed at a loading of 2% w/w into detergent composition B during its preparation and before the non-ionic surfactant was added. The silicas were dispersed in water at a concentration of 20% w/w before adding to the composition.

The viscosity and corrosion inhibition was determined for each formulation using the methods described above, and then the detergent compositions were allowed to stand for three months at ambient temperature. The tests for viscosity and corrosion inhibition were then repeated.

The results are summarised in Table V and they clearly show that the silicas used in the invention are capable of imparting to the detergent composition the desirable features of corrosion inhibition together with viscosity control over a prolonged period.

The minor changes in viscosity measured are not unusual in detergent compositions of this type, when the most noticeable movement in viscosity is always recorded over the first week of storage.

TABLE V

DETERGENT COMPOSITION	DETERGENT COMPOSITION INITIAL PREPARATION		DETERGENT COMPOSITION AFTER 3 MONTHS STANDING	
	VISCOSITY (mPas)	CORROSION WEIGHT LOSS OF Al. (g/m <sup>2</sup> /h)	VISCOSITY (mPas)	CORROSION WEIGHT LOSS OF Al. (g/m <sup>2</sup> /h)
SILICA A		792	669	0.0035
SILICA F		748	687	0.0051
SILICA G		704	739	0.0012

It must be emphasised that no physical separation was observed throughout the time period the detergent compositions were allowed to stand, showing the silicas used in the invention did not affect the physical stability of the liquid composition.

#### 5 EXAMPLE V

The detergent composition containing silica A described in Example IV, after standing for three months, was tested, together with a control containing no silica, for their effectiveness in protecting enamel from corrosion using the test method described above.

The corrosion data obtained is summarised in Table VI.

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TABLE VI

	<u>Weight Loss of Enamel</u> g/m <sup>2</sup> /h
15 Detergent Composition without silica	0.420
Detergent Composition + Silica A	0.025

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The results indicate the silicas used in the invention give adequate protection to enamel even after storage.

#### Claims

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1. Physically stable aqueous liquid detergent composition with improved non-corrosive properties and satisfactory viscosity, comprising conventional detergent ingredients characterised in that it further comprises an effective amount of amorphous silica having a surface area in the range from about 200 to about 550 m<sup>2</sup>/g, a mean pore diameter from about 1.5 to about 11 nm, an oil absorption from about 40 to about 140 cc/100g and a weight mean particle size from about 1 to about 30 micron.

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2. A detergent composition according to claim 1 wherein the silica is a precipitated silica.

3. A detergent composition according to claim 1 or 2 having a viscosity not more than about 1,000 mPas.

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4. A detergent composition according to any preceding claim wherein the silica increases the viscosity of the base composition by not more than about 50% preferably not more than 40% at a concentration of 5% w/w.

5. A detergent composition according to any preceding claim wherein the silica has a mean pore diameter of at least about 3nm.

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6. A detergent composition according to any preceding claim wherein the silica has an oil absorption of at least about 70 cc/100g.

7. A detergent composition according to any preceding claim wherein the silica has a weight mean particle size of at least about 5 micron.

8. A detergent composition according to any preceding claim wherein the silica has a weight mean particle size of up to about 20 microns.

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9. Detergent composition according to any preceding claim wherein the amorphous silica is included in an amount of from about 1 to about 5% by weight of the total composition.

10. A detergent composition according to claim 9 wherein the amorphous silica is included in an amount of from about 2 to about 4% by weight of the total composition.

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11. A liquid detergent composition according to any preceding claim, comprising from about 5 to about 30% by weight of detergent active materials of the anionic, nonionic, cationic and/or zwitterionic type, from about 5 to about 50% by weight of builder materials, characterised in that the silica is included in an amount of from about 1% to about 5% by weight of the total composition.

12. Method of preventing corrosion of machine parts during washing, characterised in that a detergent composition according to any one of the preceding claims is applied in the washing process.

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13. The use of an amorphous silica having a surface area in the range from about 200 to 550 m<sup>2</sup>/g, a mean pore diameter from about 1.5 to about 11 nm, an oil absorption from about 40 to about 140 cc/100 g and a weight mean particle size from about 1 to about 30 micron to provide a physically stable aqueous liquid detergent with improved non-corrosive properties and satisfactory viscosity.

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